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APPLICATIONS REPORT ON REPRODUCIBLE THERMISTOR REFINEMENT PROGRAM

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Electronics Branch
Manufacturing Technology Laboratory
Aeronautical Systems Division
Air Force Systems Command
United States Air Force
Wright-Patterson Air Force Base, Ohio

ASD Project Nr. 7-838

(Prepared under Contract AF 33(657)-7104 by the W. R. Grace & Co., Research Division, Clarkville, Maryland, M. C. Vanik, W. T. Barrett, J. E. Herrera, M. G. Sanchez, E. M. Glocker, S. E. Ketner, B. B. White).

NOTICES

FOREWORD

This Applications Technical Documentary Report covers all work performed under Contract AF33(657)-7104 from 5 September 1961 to 31 December 1962. The manuscript was released by the author on 28 February 1963 for publication as an ASD Technical Documentary Report.

This contract with the W. R. Grace & Co., Research Division of Clarksville, Maryland, was initiated under ASD Project 7-838, "Reproducible Thermistor Refinement Program". It was administered under the direction of Lieutenant T. Bailey, Electronics Engineer, Aeronautical Systems Division, Wright-Patterson Air Force Base, Ohio.

Dr. M. C. Vanik of W. R. Grace's Washington Research Center, Inorganic Chemical Research Department, was the supervisor in charge. Others who cooperated in the research and in the preparation of the report were: Dr. M. G. Sanchez, Director of Inorganic Chemical Research Department; Dr. W. T. Barrett, Former Director of Inorganic Chemical Research; Mr. J. E. Herrera, Chemist, Inorganic Chemical Research Department; Mr. E. M. Glocker, Manager Research Statistics, Economic Evaluation Department; Mr. S. E. Ketner, Statistician, Economic Evaluation Department and Miss B. B. White, Assistant Libraian, Research Services Department.

This project has been accomplished as a part of the Air Force Manufacturing Methods Program. The primary objective of the Air Force Manufacturing Methods Program is to develop, on a timely basis, manufacturing processes, techniques and equipment for use in economical production of USAF materials and components. The program encompasses the following technical areas:

Rolled Sheet, Forgings, Extrusions, Castings, Fiber & Power Metallurgy, Component Fabrication, Joining, Forming, Materials Removal, Fuels, Lubricants, Ceramics, Graphites, Non-Metallic Structural Materials, Solid-State Devices, Passive Devices, Thermionic Devices

Your comments are solicited on the potential utilization of the information contained herein as applied to your present or future production programs. Suggestions concerning additional Manufacturing Methods development required on this or other subjects will be appreciated.

ABSTRACT

REPRODUCIBLE THERMISTOR REFINEMENT PROGRAM

Milton C. Vanik
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W. R. Grace & Co.
Research Division

Gold-doped monocrystalline silicon exhibits temperature-resistivity behavior suitable for making highly reproducible, predictable and sensitive thermistors. Two types of thermistors were developed with reproducibilities of ±2% and operable ranges including -85 to +200°C. Manufacturing methods were developed and demonstrated on an unbalanced pilot line.

A literature survey indicated that specially doped silicon or germanium offer the best possibilities of being used as single-crystal semiconductor thermistors. Silicon was chosen for development on the basis of energy gap, purity, resistivity, and availability.

Many elements were screened as possible silicon thermistor dopants. These include gold, copper, silver, nickel, iron, zinc, platinum, manganese, and thallium. Only gold in both P- and N-type silicon gave products suitable for use in the -85 to +200°C range.

Gold-doped N-type silicon produces a high resistivity thermistor material with nearly linear $\log \rho$ vs $\frac{1}{T}$ response in the range -85°C to +200°C. This material has a temperature coefficient of resistivity of -7% per degree at 25°C. It is most suitable for high temperature, narrow range, high sensitivity applications.

Gold-doped P-type silicon is a lower resistivity thermistor material with linear $\log \rho$ vs $\frac{1}{T}$ behavior over the range of -85 to about 50°C. The temperature coefficient of resistivity of this material is -4.5% per degree at 25°C.

A statistical study was completed comparing float zone leveled with diffused gold-doped thermistor silicon. The float zone-leveling process produces the better product.

ABSTRACT

Forming of ohmic and mechanically strong contacts to the thermistor silicon is extremely important. Stability of the thermistors is very dependent upon these contacts.

Manufacturing processes encompassing 24 steps for gold-doped N-type thermistors and 28 steps for gold-doped P-type thermistors have been established. An unbalanced pilot line was set up and operated according to these processes. A demonstration run was completed for the Air Force contract officer.

Six hundred samples were produced on this pilot line and sent to the Air Force for distribution. Many of these samples are within ±1% of a standard curve. All are within contract specifications of ±2%.

These thermistors should be useful in any application which calls for precise and/or closely matched thermistors.

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PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:

JACK R. MARSH
Assistant Chief
Manufacturing Technology Laboratory
Directorate of Materials & Processes

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I INTRODUCTION

Conventional thermistors are now made from transition metal oxides by ceramic techniques. These metal oxides are semiconductors. Their electrical resistance decreases as they are heated. Dozens of other semiconductor materials could conceivably be used in place of the present thermistors as temperature-sensing devices. However, a material that is to be selected for thermistor use should have adequate sensitivity to temperature, reproducibility, and reliability.

Probably the purest and most reproducible semiconductor materials available today are the large single crystals of silicon and germanium that are used in the manufacture of electronic devices such as transistors and diodes. Silicon crystals often contain electrically active impurities as low as one ppb or less.

The objective of this project is the development of manufacturing methods and the pilot plant production of thermally sensitive devices which take advantage of the high reliability and reproducibility offered by large single-crystal semiconductors.

II DISCUSSION

I. Objectives

The main interest of this effort was to develop manufacturing methods for thermally sensitive semiconductor devices from a monocrystalline semiconductor approach. These single crystalline thermistors were to be highly reproducible, predictable and sensitive. A pilot line capability was to be developed, capable of producing as many as 4,000 units per month when operated on a balanced basis. The design and construction of the elements was to facilitate quantity production at a reasonable cost. The effort was to include an evaluation of all existing semiconductors, either single element or junction, as possible thermistor replacements and the exploration of temperature dependency in new semiconductor materials.

The desired operating range of the thermistors was to be -85°C to +200°C with the resistivity range as broad as possible. The temperature coefficient of resistance was to be comparable or larger than the TC of commercial thermistors and still satisfy the requirements. The 25°C resistance value was to be reproducible within ±2% between units of the same nominal resistance value. A temperature-resistance curve from unit to unit was to be reproducible within ±2% throughout the temperature range of -85 to +200°C. The operating life objective was 3000 hours when the elements are maintained at any static temperature within the range of -85°C to +200°C.

2. Basic Accomplishments

A literature survey indicated that specially doped silicon or germanium offer the best possibility of being used as a single-crystal semiconductor thermistor. Silicon was chosen for development on the basis of energy gap, purity, resistivity and availability. Many elements were screened as possible silicon dopants; these include gold, copper, silver, nickel, iron, zinc, platinum, manganese, and thallium. Only gold in both P- and N-type silicon gave products suitable for use in the -85 to +200°C range.

Gold-doped N-type silicon produces a high resistivity thermistor material with nearly linear log ρ vs 1/T response in the range of -85 to +200°C. This material has a temperature coefficient of resistivity of -7% per degree at 25°C. It is most suitable for high temperature, narrow range, high sensitivity applications. Gold-doped P-type silicon is a lower resistivity thermistor material with nearly linear log ρ vs 1/T behavior over the range of -85 to about 50°C. The temperature coefficient of resistivity of this material is -4.5% per degree at 25°C.

A statistical study was completed comparing float leveled with the diffused gold-doped thermistor silicon. The float-zone leveling process produces a better product.

Forming of ohmic and mechanically strong contacts to the thermistor silicon is extremely important. Stability of thermistors is very dependent upon the contacts. In the case of P-type silicon, contacts were made by vacuum depositing aluminum on the gold-doped silicon, alloying the aluminum into the silicon at elevated temperatures, and then nickel-plating and soldering the leads. Contacts to N-type gold-doped silicon thermistors were made by either simple nickel-plating or with subsequently soldering of the leads, or by first applying a sub-layer of antimony, alloying, nickel-plating, and soldering the leads.

Manufacturing processes encompassing 24 steps for gold-doped N-type thermistors and 28 steps for gold-doped P-type thermistors have been established. An unbalanced pilot line was set up and operated according to the processes. A demonstration run was completed for the Air Force Contract Officer. Six hundred samples were produced on this pilot line and sent to the Air Force for distribution. Many of the samples were within ±1% of the standard curve. All within the contract specifications of ±2%.

3. <u>Manufacturing Process for Gold-Doped P-Type Silicon</u>

A 28-step manufacturing process was developed for producing gold-doped P-type silicon thermistors. The unbalanced pilot line was operated according to this process while producing the 400 contract samples of P-type thermistors. The 28 steps are outlined below:

1. Determine nesessary starting silicon resistivity.
2. Purchase monocrystalline rod from a commercial supplier, or prepare the same.
3. Check resistivity profile with 4-point probe.
4. Prepare rod for zone leveling: Etch 1:3 HF/HNO_3, water dry.
5. Zone level with high-purity gold.
6. Cut three wafers from seed end, chuck end, and center.
7. Prepare three wafer-size thermistors from these slices and measure resistance at 25°C.
8. Calculate desired thickness of production wafers basis resistivity of the three trial wafers and dicing tool sizes.
9. Cut remaining zone-leveled rod into wafers.
10. Wash with alcohol, acetone, alcohol.
11. Lap wafers with 180-C silicon carbide paper.
12. Wash with water, wipe with paper to remove fine particles.
13. Wash successively with acetone-alcohol-water-HF-water-alcohol.
14. Vacuum deposit aluminum on wafers in vacuo.
15. Alloy aluminum to silicon at 580-630°C.
16. Etch wafers with concentrated HF-water.
17. Wipe with paper, wash with water-HF-water.
18. Immerse wafers briefly in boiling ammonium hydroxide.
19. Nickel plate wafers by electrodeless technique at 96°C.
20. Heat treat at 210°C overnight in argon or nitrogen.
21. Recheck resistivity of several wafers with pressure contacts.
22. Determine final dice size.
23. Dice wafers.
24. Solder leads to dice with tin at 240°C-250°C.
25. Wash with methyl alcohol.
26. Adjust size of thermistors by high-speed diamond grinding comparing resistance to resistance of a standard thermistor.
27. Determine final temperature-resistance behavior at a mininum of three temperatures.
28. Paint and bake at 210°C.

Although some short cuts might be made in the process we feel the most consistent results will be obtained by following the described procedure.

4. <u>Manufacturing Process for Gold-Doped N-Type Silicon</u>

A 24-step manufacturing process was developed for producing gold-doped N-type thermistors. The unbalanced pilot line was operated according to this process while producing the 200 contract samples of N-type thermistors. The 24 steps are outlined below:

1. Determine necessary starting silicon resistivity.
2. Purchase monocrystalline rod from a commercial supplier, or prepare the same.
3. Check resistivity profile with 4-point probe.
4. Prepare rod for zone leveling: Etch 1:3 HF/HNO_3, dry.
5. Zone level with high-purity gold.
6. Cut three wafers from seed end, chuck end, and center.
7. Prepare three wafer-size thermistors from these slices and measure resistance at 25°C.
8. Calculate desired thickness of production wafers based on resistivity of the three trial wafers and dicing tool sizes.
9. Cut remaining zone-leveled rod into wafers.
10. Wash with alcohol, acetone, alcohol.
11. Lap wafers with 180-C silicon carbide paper.
12. Wash with water, wipe with paper to remove fine particles.
13. Wash successively with acetone-alcohol-water-conc. HF-water.
14. Immerse wafers briefly in boiling ammonium hydroxide.
15. Nickel plate wafers by electrodeless technique at 96°C.
16. Heat treat at 210°C overnight in argon or nitrogen.
17. Recheck resistivity of several wafers with pressure contacts.
18. Determine final dice size.
19. Dice wafers.
20. Solder leads to dice with tin at 240°C.
21. Wash with methyl alcohol.
22. Adjust size of thermistors by high-speed diamond grinding comparing resistance to resistance of a standard thermistor.
23. Determine final temperature-resistance behavior at a minimum of three temperatures.
24. Paint and bake at 210°C.

Again, we feel that although some short cuts might be made in this process, the most consistent results will be obtained by following the described procedure.

5. Description of Devices

Figure 1 is a photograph of some of the typical thermistors prepared under the contract. These devices consist essentially of a small piece of gold-doped silicon sandwiched between an electrode consisting of a sub-layer of aluminum or antimony and a cover layer of nickel plating to which leads are soldered with tin. The entire device, with the exception of the leads, is coated with a white silicone enamel for electrical insulation properties and also to prevent photoconductive effects in the gold-doped silicon. Samples prepared under the contract were three general sizes and types. Gold-doped P-type samples were prepared with resistances at 25°C of 4000 ohm, 5000 ohm and 500 ohm. The four and five thousand ohm samples were cylindrical in shape, being approximately 2.5 mm in diameter by 2 mm in length. The 500 ohm P-type samples measured approximately 3.8 x 3.8 x 2.3 mm, the electrode area being square. These were the largest thermistors produced and had the most undesirable time constants of response. However, they were the easiest to adjust to a given resistance, for the larger the size the easier it is to adjust the thermistors to within ±2% of a reference resistance. The gold-doped N-type silicon thermistors made under the contract were 500k ohms at 25°C and measured 2x2x1 mm. Again, the electrodes were square and the dice were made by cross-cutting techniques on the high-speed diamond saw. Of course, smaller sizes can be made with present dicing techniques. However, it should be pointed out that size adjustment becomes increasingly difficult on the smaller sizes. Since the coefficient of thermal conductivity of metallic silicon is about 70 times greater than iron oxide, a silicon thermistor device with a time constant of 2 seconds can be somewhat larger in size than an iron oxide-type thermistor with a 2 second time constant.

FIGURE 1

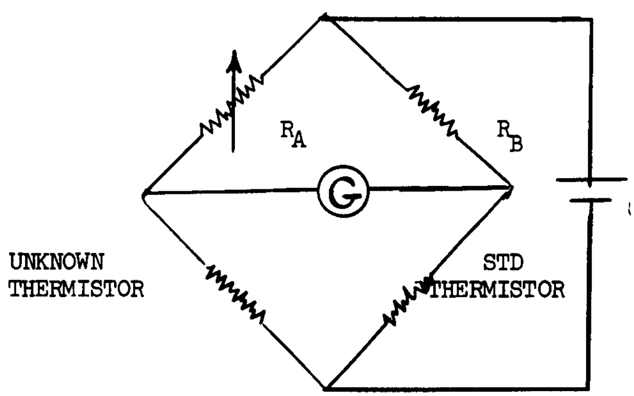
Finished thermistors, actual size. Comparison is made with a Bendix ML419/AMT-4 thermistor.

6. Test Equipment and Procedures

Essentially there were no new test equipment or procedures used in testing of the thermistors made on the unbalanced pilot line. Resistances were measured with a standard Wheatstone Bridge. Standard temperature-resistance curves were established by measuring many thermistors at closely spaced intervals over the entire working range of the thermistor. These data were programmed into a computer which calculated the least square curves and typed out standard curves for the thermistors at 1 degree intervals over the operating range. On P-type silicon the slope as calculated by the computer was usually within ±0.0015 electron volts of the average slope. The constant-temperature baths in which these measurements were made were held to within ±0.01°C. The thermistors were mounted in these baths in a U-shaped Teflon tube with a small amount of silicone oil at the bottom covering the thermistor.

After good standard curves had been established for the various types of silicon, it was possible to determine whether a thermistor was within ±2% of this curve by measuring the resistance of the thermistor at 3 points on equally spaced intervals within the operating range. In no case was a thermistor measured at 3 points and found to be within ±2% specs, later found to be out of specs by measuring at other points within the temperature range. For this reason, it was felt that 3 points at equally spaced intervals over the temperature range could be considered to be sufficient for determining whether a thermistor was within the proper temperature-resistance specifications.

Another measuring technique, which was used after good standard curves had been established, is the ratio technique of measuring resistances. An unknown thermistor and a standard thermistor are mounted in opposite arms of a Wheatstone bridge.



$$\frac{R_{UNK}}{R_{STD}} = \frac{R_A}{R_B}$$

FIGURE 2

Both thermistors are immersed in the same constant-temperature bath. The reading obtained is the ratio of the resistance of the unknown to the standard resistance. Standards are chosen so the ratio will be approximately equal to one and so that the standard fits the standard curve for a group of thermistors very accurately. Under these circumstances when the ratio goes beyond 1.02 or below 0.98, a thermistor can be considered to be out of spec at that particular temperature. It is not absolutely necessary to know the absolute temperature of a particular constant-temperature bath. One can then be satisfied with being within several degrees of a desired testing temperature. Since the time constants of the thermistors are usually small compared to the time of temperature cycling of the bath, effects due to temperature fluctuation in the baths tend to be eliminated because these will not appreciably affect the ratio. We found that this technique improved our measurement precision considerably and also speeds up one of the most time-consuming steps in the manufacturing process, that of measuring the final temperature resistance properties.

7. Availability

The unbalanced pilot line which was established to meet the contract requirements is available for production of the precise gold-doped silicon thermistors if there is sufficient interest to warrant its use. This line if fully staffed is capable of producing as many as 4000 units per month. Presently the lead time on an order consisting of several-hundred thermistors which are not in stock would be on the order of 4 to 6 weeks. That is, delivery could be expected within 4 to 6 weeks of receipt of the order.

Six hundred samples of various types mentioned have been prepared and shipped to the Air Force for distribution. Samples may be obtained by writing to:

Lt. D. Fitzgerald
Aeronautical Systems Division (ASRCTE)
United States Air Force
Wright-Patterson Air Force Base, Ohio
U.S.A.

8. <u>Temperature-Resistance Curves</u>

We are including in Tables 1, 2, 3, and 4, temperature-resistance curves for the various samples that were supplied to the Air Force for distribution. Each thermistor is packed with a standard curve of this nature and can be expected to be within ±2% of the specified resistances at any temperature in the operating range. The temperature-resistance curves cover temperatures from -85 to +200°C. The gold-doped P-type thermistors are not calibrated above 30°C since they tend to lose linearity and deviate from a straight line. However, these thermistors will withstand an extended aging period at 200°C without change in the specified standard-resistance curve. We also feel that this curve can be extended above 30°C with special fitting techniques.

TABLE 1

Gold-Doped P-Type Silicon

Temperature vs. Resistance -85°C. to +30°C.

Temp. °C.	Res. Ω	Temp. °C.	Res. Ω	Temp. °C.	Res. Ω
-85	1097K	-45	27.02K	- 5	2082
-84	980.9K	-44	25.05K	- 4	1974
-83	878.0K	-43	23.24K	- 3	1872
-82	786.9K	-42	21.58K	- 2	1776
-81	706.0K	-41	20.05K	- 1	1685
-80	634.1K	-40	18.64K	0	1600
-79	570.2K	-39	17.33K	1	1520
-78	513.3K	-38	16.14K	2	1445
-77	462.6K	-37	15.03K	3	1374
-76	417.3K	-36	14.01K	4	1307
-75	376.9K	-35	13.06K	5	1243
-74	340.7K	-34	12.19K	6	1184
-73	308.4K	-33	11.38K	7	1127
-72	279.3K	-32	10.63K	8	1074
-71	253.3K	-31	9941	9	1024
-70	229.9K	-30	9298	10	976.0
-69	208.9K	-29	8703	11	931.0
-68	190.0K	-28	8150	12	888.4
-67	172.9K	-27	7636	13	848.0
-66	157.5K	-26	7159	14	809.9
-65	143.7K	-25	6716	15	773.7
-64	131.1K	-24	6303	16	739.4
-63	119.8K	-23	5919	17	706.9
-62	109.5K	-22	5561	18	676.1
-61	100.2K	-21	5228	19	646.9
-60	91.80K	-20	4917	20	619.1
-59	84.15K	-19	4627	21	592.8
-58	77.20K	-18	4357	22	567.8
-57	70.88K	-17	4104	23	544.0
-56	65.14K	-16	3868	24	521.5
-55	59.90K	-15	3648	25	500.0
-54	55.13K	-14	3441	26	479.6
-53	50.78K	-13	3248	27	460.2
-52	46.81K	-12	3068	28	441.7
-51	43.18K	-11	2898	29	424.1
-50	39.86K	-10	2740	30	407.3
-49	36.83K	- 9	2591		
-48	34.05K	- 8	2452		
-47	31.50K	- 7	2321		
-46	29.17K	- 6	2198		

TABLE 2

Gold-Doped P-Type Silicon

Temperature vs. Resistance -85°C. to +30°C.

Temp. °C.	Res. Ω	Temp. °C.	Res. Ω	Temp. °C.	Res. Ω
-85	8525K	-45	212.9K	- 5	16.57K
-84	7625K	-44	197.4K	- 4	15.71K
-83	6828K	-43	183.2K	- 3	14.90K
-82	6122K	-42	170.1K	- 2	14.14K
-81	5495K	-41	158.1K	- 1	13.42K
-80	4938K	-40	147.0K	0	12.75K
-79	4442K	-39	136.8K	1	12.11K
-78	4000K	-38	127.4K	2	11.51K
-77	3606K	-37	118.7K	3	10.95K
-76	3255K	-36	110.6K	4	10.41K
-75	2940K	-35	103.2K	5	9911
-74	2659K	-34	96.33K	6	9437
-73	2407K	-33	89.96K	7	8989
-72	2182K	-32	84.07K	8	8566
-71	1979K	-31	78.61K	9	8166
-70	1797K	-30	73.55K	10	7787
-69	1633K	-29	68.86K	11	7429
-68	1486K	-28	64.50K	12	7091
-67	1353K	-27	60.45K	13	6770
-66	1233K	-26	56.69K	14	6466
-65	1125K	-25	53.19K	15	6179
-64	1027K	-24	49.93K	16	5906
-63	938.5K	-23	46.90K	17	5648
-62	858.4K	-22	44.08K	18	5402
-61	785.8K	-21	41.45K	19	5170
-60	719.9K	-20	38.99K	20	4949
-59	660.2K	-19	36.70K	21	4739
-58	605.8K	-18	34.56K	22	4540
-57	556.5K	-17	32.57K	23	4351
-56	511.5K	-16	30.70K	24	4171
-55	470.5K	-15	28.96K	25	4000
-54	433.2K	-14	27.33K	26	3837
-53	399.1K	-13	25.80K	27	3683
-52	368.0K	-12	24.37K	28	3535
-51	339.6K	-11	23.03K	29	3395
-50	313.6K	-10	21.77K	30	3261
-49	289.8K	- 9	20.60K		
-48	268.0K	- 8	19.49K		
-47	248.0K	- 7	18.45K		
-46	229.7K	- 6	17.48K		

TABLE 3

Gold-Doped P-Type Silicon

Temperature vs. Resistance -85°C. to +30°C.

Temp. °C.	Res. Ω	Temp. °C.	Res. Ω	Temp. °C.	Res. Ω
-85	10656K	-45	266.1K	- 5	20.71K
-84	9531K	-44	246.8K	- 4	19.63K
-83	8535K	-43	229.0K	- 3	18.62K
-82	7652K	-42	212.7K	- 2	17.67K
-81	6868K	-41	197.6K	- 1	16.77K
-80	6172K	-40	183.8K	0	15.93K
-79	5552K	-39	171.0K	1	15.14K
-78	5000K	-38	159.2K	2	14.39K
-77	4508K	-37	148.3K	3	13.68K
-76	4068K	-36	138.3K	4	13.02K
-75	3676K	-35	129.0K	5	12.39K
-74	3324K	-34	120.4K	6	11.80K
-73	3009K	-33	112.5K	7	11.24K
-72	2727K	-32	105.1K	8	10.71K
-71	2474K	-31	98.27K	9	10.21K
-70	2246K	-30	91.94K	10	9734
-69	2041K	-29	86.07K	11	9287
-68	1857K	-28	80.62K	12	8863
-67	1691K	-27	75.56K	13	8463
-66	1541K	-26	70.86K	14	8083
-65	1406K	-25	66.48K	15	7723
-64	1284K	-24	62.41K	16	7383
-63	1173K	-23	58.63K	17	7060
-62	1073K	-22	55.10K	18	6753
-61	982.2K	-21	51.81K	19	6462
-60	899.9K	-20	48.74K	20	6186
-59	825.2K	-19	45.88K	21	5924
-58	757.3K	-18	43.21K	22	5675
-57	695.6K	-17	40.71K	23	5439
-56	639.4K	-16	38.38K	24	5214
-55	588.1K	-15	36.20K	25	5000
-54	541.5K	-14	34.16K	26	4797
-53	498.9K	-13	32.25K	27	4603
-52	460.0K	-12	30.46K	28	4419
-51	424.5K	-11	28.79K	29	4244
-50	392.0K	-10	27.22K	30	4077
-49	362.3K	- 9	25.75K		
-48	335.0K	- 8	24.36K		
-47	310.1K	- 7	23.07K		
-46	287.1K	- 6	21.85K		

TABLE 4

Gold-Doped N-Type Silicon

Temperature vs. Resistance 0°C. to 200°C.

Temp. °C.	Res. Ω	Temp. °C.	Res. Ω	Temp. °C.	Res. Ω	Temp. °C.	Res. Ω
0	3320K	51	96.02K	102	7498	153	1104
1	3057K	52	90.61K	103	7183	154	1069
2	2816K	53	85.53K	104	6884	155	1035
3	2596K	54	80.77K	105	6599	156	1002
4	2395K	55	76.30K	106	6327	157	969.8
5	2211K	56	72.11K	107	6067	158	939.1
6	2042K	57	68.17K	108	5820	159	909.6
7	1887K	58	64.46K	109	5584	160	881.1
8	1744K	59	60.98K	110	5359	161	853.7
9	1614K	60	57.71K	111	5144	162	827.2
10	1494K	61	54.63K	112	4938	163	801.7
11	1384K	62	51.74K	113	4742	164	777.0
12	1282K	63	49.01K	114	4555	165	753.3
13	1189K	64	46.44K	115	4376	166	730.4
14	1103K	65	44.02K	116	4205	167	708.3
15	1024K	66	41.75K	117	4042	168	686.9
16	950.8K	67	39.60K	118	3885	169	666.3
17	883.4K	68	37.57K	119	3736	170	646.4
18	821.3K	69	35.66K	120	3593	171	627.2
19	764.0K	70	33.86K	121	3456	172	608.7
20	711.0K	71	32.16K	122	3325	173	590.8
21	662.0K	72	30.55K	123	3200	174	573.5
22	616.7K	73	29.04K	124	3080	175	556.7
23	574.7K	74	27.60K	125	2965	176	540.6
24	535.9K	75	26.25K	126	2855	177	525.0
25	500.0K	76	24.97K	127	2750	178	509.9
26	466.7K	77	23.76K	128	2649	179	495.3
27	435.8K	78	22.61K	129	2552	180	481.2
28	407.1K	79	21.53K	130	2460	181	467.5
29	380.5K	80	20.50K	131	2371	182	454.3
30	355.9K	81	19.53K	132	2286	183	441.6
31	332.9K	82	18.61K	133	2204	184	429.2
32	311.6K	83	17.74K	134	2125	185	417.2
33	291.8K	84	16.91K	135	2050	186	405.7
34	273.3K	85	16.13K	136	1978	187	394.5
35	256.2K	86	15.38K	137	1909	188	383.7
36	240.2K	87	14.68K	138	1842	189	373.2
37	225.3K	88	14.01K	139	1778	190	363.0
38	211.4K	89	13.38K	140	1717	191	353.2
39	198.5K	90	12.77K	141	1658	192	343.6
40	186.4K	91	12.20K	142	1601	193	334.4
41	175.2K	92	11.66K	143	1547	194	325.5
42	164.7K	93	11.14K	144	1494	195	316.8
43	154.8K	94	10.65K	145	1444	196	308.4
44	145.7K	95	10.19K	146	1395	197	300.3
45	137.1K	96	9742	147	1349	198	292.4
46	129.1K	97	9321	148	1304	199	284.8
47	121.6K	98	8919	149	1261	200	277.3
48	114.5K	99	8537	150	1220		
49	108.0K	100	8174	151	1180		
50	101.8K	101	7827	152	1141		

9. <u>Time Constant of Response</u>

We define the time constant of response as the time required for the thermistor to indicate 63% of the total increment in temperature in a stream of air with a speed of 820 feed per minute. This is essentially the U.S. Weather Bureau definition found in their specification for radiosonde thermistors. The increment in temperature can be positive or negative and the results should be about the same. The factor being measured in this test is the ability of a thermistor to accept or dispose of heat to its environment.

To measure the time constant a small wind tunnel was made of 2 air intakes whose temperature and speed could be independently regulated. Temperatures used were 25°C and -10°C for testing the P-type material and the Bendix AMT4/ML419 radiosonde thermistor; for the N-type, 90°C and 25°C. The speed of the air was 820 feet per minute. Ten readings were taken for each sample. For a given thermistor, or a group of thermistors of the same kind, the maximum variation in the measurement was 0.4 seconds. The following table summarizes the results.

TABLE 5

Thermistor Number	Size in mm	Ohms at 25°C	Time Constant in Seconds Minimum(1)	Maximum(1)
2m-3-7	2.5 dia. x 2.5	4K	2.0	2.2
W5-35	2.6 dia. x 2.6	4K	2.1	2.4
W2-34	2.6 dia. x 2.3	4K	2.1	2.2
3253-A-10	2.2 x 2.2 x 2.0	500K	2.1	2.4
3253-A-14	2.2 x 2.3 x 2.2	500K	2.1	2.4
3253-A-16	2.3 x 2.3 x 2.3	500k	2.3	2.5
2M-2-11	2.5 dia. x 3.0	5K	2.9	3.1
2M-2-21	2.5 dia. x 2.9	5K	2.9	3.3
2M-3-15	2.5 dia. x 2.9	5K	3.1	3.3
W-14-8	3.7 dia. x 1.9	.5K	2.9	3.2
Bendix M1-419	.7 dia. x 39	50K	3.1	3.4
W-15-4	3.8 x 3.8 x 2.3	.5K	4.2	4.5
W-6-12	7.8 x 3.8 x 2.5	.5K	4.7	5.1

(1) Average of ten readings.

This table shows that the time constant of our thermistors except the bulky 3.8 x 3.8 x 2.3 ml size is equivalent to, equal, or better than, the time constant of the Bendix thermistor and within the specifications of 5 seconds for a radiosonde thermistor.

10. Dissipation Constant

The dissipation constant is defined as the amount of power in milliwatts that has to be applied to a thermistor to raise the temperature of the thermistor one degree centigrade above the surrounding medium. For surrounding medium, we specify well stirred oil baths and still air. Both values are given with the characterization chart of the thermistor.

To measure the dissipation constant of the thermistors in still air, we used a cardboard box with a capacity of about 25 liters. The thermistors were evenly distributed, hanging from the top of the box by their own leads. The box was sealed. Then the resistance of all the thermistors was measured using a current through the sample of 5 microamps. The resistance was remeasured increasing the amount of current. This step was repeated until the resistance of every thermistor showed a decrease equivalent to one degree centigrade or more. The measurements in oil were made using Dow Corning 200-20 silicone oil stirring at constant speed and at 25°C. Again several measurements were taken increasing the current through the thermistor before each measurement until the resistance showed a decrease equivalent to 1° centigrade or more.

With the values obtained in the previous tests we were able to draw a smooth curve for each thermistor and find the exact value of the current that will give the difference of 1° centigrade higher than the surrounding medium. Having the current "I" and the resistance "R" of the thermistor, the power in milliwatts was calculated using the formula: $W = I^2R$. The following table summarizes the results.

TABLE 6

Thermistor Number	Size in mm	Ohms at 25°C	Dissipation Factor: in still air (milliwatt/°C)	Dissipation Factor: In a well stirred oil bath (milliwatt/°C)
W-6-12	3.8 x 3.8 x 2.5	0.5K	3.7	54
W-14-8	3.7 dia. x 1.9	0.5K	2.6	37
W-15-4	3.8 x 3.8 x 2.3	0.5K	2.6	-
2mm-3-15	2.5 dia x 2.9	5K	2.8	52
2mm-2-11	2.5 dia x 3.0	5K	2.8	60
W-5-35	2.6 dia x 2.6	4K	2.2	35
W-2-34	2.6 dia x 2.3	4K	2.1	35
2mm-3-7	2.5 dia x 2.5	4K	1.9	37
Bendix MI-419	0.7 dia x 39	50K	0.8	1.5
I-27	2.2 x 2.2 x 2.2	500K	0.4	8
I-22	2.2 x 2.3 x 2.2	500K	0.4	8
A-14	2.2 x 2.3 x 2.2	500K	0.4	7

This chart shows first, a fairly good reproducibility for thermistors of the same size and range; second, that the dissipation constant for the same thermistor is about 20 times higher in the oil bath than it is in air; and third, that all our thermistors, excepting perhaps the high resistance, small size, N-type, have a higher dissipation constant than the Bendix thermistor.

11. Static Temperature Lifetime

Gold-doped silicon thermistors appear to be quite stable at any temperature in the operating range. It is felt that the most severe conditions are those that occur at the upper end of the suggested operating range of 200°C. Any thermistor which is stable at 200°C would be stable at any temperatures below 200°C. The table below summarizes aging data at 200°C for various types of thermistors both in the wafer size and in the diced form.

TABLE 7

Thermistor Stability

Thermistor No.	Type	Original Resistance (Ohms)	Hours at 200°C	Resistance After Treatment at 200°C.
1	N	7305	1765	7302
2	N	7345	1765	7345
3	N	5688	1765	5685
4	N	5641	1765	5636
5	P	19.1	2199	18.8
6	P	22.1	2199	22.0
7	P	19.2	2199	19.1
8	P	18.6	2199	18.6
9	N	10,490	2816	10,150
10	N	12,230	2816	12,090
11	N	12,650	2816	12,460
12	P	635	4934	633
13	P	611	4934	615
14	P	614	4934	614
15	P	606	4934	603
16	P	610	4934	608

These data demonstrate that both the P- and N-type thermistor material are stable for extended periods of time, on the order of several thousand hours at temperatures in the range of 200°C. Frequently the change in resistivity is little or less than 1%.

12. Fitting of Experimental Points by Empirical Equation

The curves for both P- and N-type gold-doped silicon can deviate from a straight line on a logarithm of resistivity versus reciprocal of the absolute temperature plot. This deviation is greater in the case of P-type silicon, becoming significant at 25 or 30°C. At higher temperatures the curve flattens out such that at 200°C there is little sensitivity to temperature. However, there is still some sensitivity to temperature at, say, 100°C. We felt that if the experimental points could be fitted by a carefully selected empirical equation, it would extend the predictable portion of the curve. Hence the thermistors might be useable to 100°C, admittedly with some loss of sensitivity.

Equations for the resistivity as a function of temperature have been derived for gold-doped silicon. These have the general form:

$$R = B_1T^F + B_2 \exp \Delta E/_{KT}$$

where:

R = resistance in ohms

B_1 = a constant

B_2 = a constant

F = a constant

ΔE = a constant

T = absolute temperature in °K

K = Boltzman constant

The experimental data were processed through a computer to find the values of the constants which gave the best fit or smallest standard deviation. In the case of the P-type silicon studied the best fit was obtained when, $\Delta E = 0.340$ e.v. and $F = 0$. The data are summarized in Table 8.

TABLE 8

Temperature (°C)	Experimentally Determined Resistance	Resistance Calculated $R = B_1 + B_2 e^{\frac{\Delta E}{KT}}$
-58.56	79,600	80,016
-50.80	42,250	42,087
-40.49	19,200	19,157
-30.91	9,926	9,807
-20.38	5,052	4,993
-10.81	2,872	2,842
.00	1,600	1,587
+ 9.92	980.0	973.8
+19.99	620.5	620.4
+30.00	411.7	414.0
+40.00	285.6	288.7
+50.03	207.5	210.1
+60.18	157.0	159.1
+70.47	124.5	125.4
+79.98	104.2	104.2
+90.35	88.5	88.4
+99.99	78.0	78.0

Standard deviation, σ, = 0.8%

$B_1 = 45.85$
$B_2 = 8.069 \times 10^{-4}$

The fit of the data is very good. In only a few cases do the calculated points vary more than 1% from the observed points. The value of $\Delta E = 0.340$ agrees well with the values determined by independent techniques (1).

The reason for deviation from a linear log ρ vs. 1/T plot appears to be due to the saturation of the gold levels at high temperatures. The value of the empirical equation is that it allows the expression of resistance and sensitivity in an explicit manner at temperatures up to 100°C for P-type silicon. It also establishes the gold donor level precisely at 0.340 e.v. above the valence band.

III CONCLUSIONS AND RECOMMENDATIONS

Single-crystal semiconductor silicon, because of its purity, resistivity, energy gap, and availability, is an outstanding candidate for monocrystalline-thermistor development.

Gold-doped monocrystalline silicon can produce thermistors which are highly reproducible, predictable and sensitive over the range of -85° to +200°C.

Float-zone leveling produces a product with less variability than diffusion material.

Forming of ohmic and mechanically sound contacts to gold-doped silicon results in good thermistor stability.

Measurement precision and speed are improved by the use of the comparison or ratio technique.

Final size adjustment is necessary to obtain high yields of devices with ±2% specifications.

Size adjustment on small thermistors is difficult.

Short-term annealing at 210°C can increase the temperature stability of gold-doped silicon.

These thermistors should be useful in any application which calls for precise or closely matched thermistors.

Yields of 85% or better are possible when using the prescribed 28-step process for P-type silicon and 24-step process for N-type silicon.

Future work should be directed toward smaller-sized thermistors with very fast response times.

Size adjusting could be improved or revised so that it can be used on very small samples.

Other techniques of encapsulation such as canning should be investigated.

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2. Thermistors-Applications
3. Thermistors-Properties
4. Temperature-Sensitive Elements
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6. Silicon Crystals

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